

# A Ruggedness Evaluation of Procedures for Damage Threshold Testing Optical Materials

Matthew W. Hooker and Milfred E. Thomas Science and Technology Corporation • Hampton, Virginia

Stephanie A. Wise and Nina D. Tappan Langley Research Center • Hampton, Virginia The use of trademarks or names of manufacturers in this report is for accurate reporting and does not constitute an official endorsement, either expressed or implied, of such products or manufacturers by the National Aeronautics and Space Administration.

This publication is available from the following sources:

NASA Center for AeroSpace Information 800 Elkridge Landing Road Linthicum Heights, MD 21090-2934 (301) 621-0390 National Technical Information Service (NTIS) 5285 Port Royal Road Springfield, VA 22161-2171 (703) 487-4650

## **Abstract**

A ruggedness evaluation of approaches to damage threshold testing was performed to determine the influence of three procedural variables on damage threshold data. In this work, the differences between the number of test sites evaluated at an applied fluence level (1 site versus 10 sites), the number of laser pulses at each test were all found to significantly influence the damage threshold data over a 99-percent confidence interval.

### Introduction

# **Damage Threshold Testing**

Laser-induced damage to optical materials has been the subject of significant research since the early 1960's, and the history of this topic is summarized by Wood (ref. 1). Laser-induced damage to an optical material is defined as the cracking, melting, or pitting of an optic or optical coating due to interaction with a high-energy laser beam. The minimum applied laser energy level that will result in damage is referred to as the damage threshold and is often the critical limitation in the selection of materials for use in high-energy laser systems.

Damage threshold testing is a controlled testing procedure used to determine the minimum applied laser fluence at which an optic will be damaged. The damage threshold of optical materials is generally determined by using one of two procedures.

The most widely accepted procedure involves the systematic exposure of a predetermined number of sites on an optic to known laser fluences. In this procedure, several spots on an optic are exposed to an incident beam at a known fluence, as shown in figure 1. The fluence level is then increased, and another set of controlled exposures is performed. This process is repeated at several fluence levels to ensure the accuracy of the data as well as the uniformity of the optic (or coating) under evaluation (refs. 1-6). Once all the planned exposures have been performed, a plot of the percentage of damaged sites observed at a given fluence versus the applied fluence is prepared. The resulting graph shows the probability of failure at a given fluence level, and the highest applied fluence at which no damage is observed is referred to as the damage threshold. An example of a plot of the probability of failure as a function of the applied laser fluence is shown in figure 2.

Another procedure to determine the damage threshold of an optical material involves the incremental increase of a laser fluence on adjacent test sites until damage is induced. Once the damage threshold has been approximated, iterative increases and/or decreases in

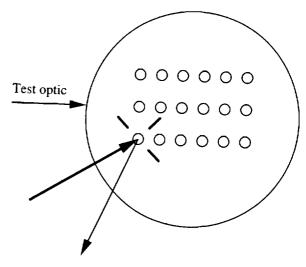


Figure 1. Test pattern for performance of damage threshold test.

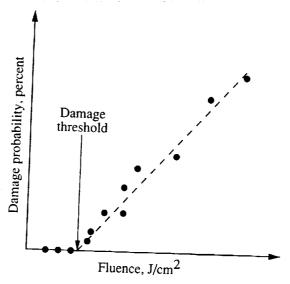


Figure 2. Representative graph showing probability of inducing optical damage as a function of applied fluence.

laser fluence are employed to determine the damage threshold of the optic. This procedure is not as structured as the previously described process but is particularly useful when the available testing area is limited (ref. 2).

# Sources of Variability in Damage Testing

The damage threshold of optical components is dependent upon several operational parameters, including the wavelength  $\lambda$ , pulse length, and polarization of the incident laser beam used for testing. Additionally, recent interlaboratory tests (refs. 7-10) have shown that the damage threshold of optical components is also dependent upon several procedural variables. In this round-robin study, similar optics were distributed to eight damage-testing facilities for testing at  $\lambda = 1.06 \mu m$ . Each facility performed damage threshold tests on the specimens and documented the operational conditions under which the data were obtained. The damage thresholds reported by the different laboratories were found to vary greatly because of the differences in the procedures employed at the various testing facilities. Besides the cleaning and handling procedures employed, procedural variables found to influence damage threshold data included the number of sites tested on a single optic (a), the number of laser pulses each test site was exposed to (b), and the diameter of the incident beam (c).

# Ruggedness Testing

Ruggedness testing (ref. 11) is a procedure for evaluating the resistance of a test protocol to bias due to variations in the test procedure. This evaluation technique employs Plackett-Burman experimental designs, which permit the evaluation of N-1 variables with N experiments. The ability to evaluate variables with a minimal number of experiments makes Plackett-Burman designs attractive for the evaluation of laboratory test procedures, as compared with other experimental designs. For example, the use of full factorial experiments would require the completion of  $2^N$  experiments, making the performance of a full factorial experiment a time and labor intensive endeavor. The experimental matrix for a threefactor ruggedness experiment is shown in table I. In this table, the plus (+) and minus (-) signs refer to the high and low experimental levels under investigation.

Table I. Design of Three-Factor Ruggedness Test

a	b	С	Trial 1	Trial 2
+	+	-		
_	-	-		
-	+	+		'
+	<u> </u>	+		
	a	a b + + + +	a b c + + -  - + + + - +	a         b         c         Trial 1           +         +         -           -         -         -           -         +         +           +         -         +

As shown in table I, each procedural variable under investigation is evaluated at two experimental levels, and the significance of the difference between the data obtained at each experimental level is evaluated by using t-tests. Once all the experimental data have been gathered, the value for  $t_N$ -1 (t at N-1 degrees of freedom) is calculated from the relation

$$t_{N-1} = \frac{\text{Average effect}}{2\sqrt{\left[\sum d_i^2/(N-1)\right]/(N/8)/\sqrt{(2N)}}}$$
(1)

where d is the difference between effects and N is the total number of experiments performed. Once  $t_{N-1}$  is calculated for each of the three variables, the calculated value is compared with the critical value for t (ref. 12). If the calculated value for  $t_{N-1}$  exceeds the critical value for t, a significant difference is said to exist between the two experimental levels. However, if  $t_{N-1}$  is less than the critical value for t, the procedure is said to be "rugged" against that operational parameter (ref. 11).

Ruggedness tests are particularly useful in developing test procedures that must be employed in more than one laboratory. A second laboratory may not possess the same biases as the first, making the data obtained at the two facilities different. By identifying all sources of variability at the development laboratory, these biases may be eliminated, thereby smoothing the transition of a new test procedure into other laboratories (refs. 13–14).

# **Experimental Procedure**

# **Test Configuration**

The damage threshold tests were performed at  $\lambda = 532$  nm with the second harmonic of a Q-switched neodymium doped yttrium aluminum garnet (Nd:YAG) laser (Continuum model NY-61) with a pulse length of  $\approx 7.5$  nsec. To perform these tests, the incident beam was directed toward the test optic as shown in figure 3.

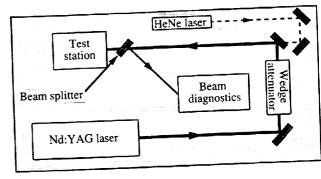


Figure 3. Layout of optics table for damage testing.

The output energy of the Nd:YAG laser was controlled with a wedge attenuator and measured using a Molectron model 5200 power meter. Additionally, the beam diameter and the spatial profile of the beam were obtained by sampling 4 percent of the incident beam with a 7° wedge, directing that portion of the beam to a Cohu solid-state camera, and using BEAMCODE diagnostic software (Big Sky Laser, Bozeman, MT). The Molectron power meter is accurate to within 0.5 percent, and the BEAMCODE diagnostic system (including the Cohu camera) is accurate to within 3 percent.

All the damage threshold tests were performed with the incident beam normal to the optic being tested, and the incident beam possessed a Gaussian fit of 94 percent (±1 percent). After all the diagnostic information describing the incident beam was obtained, the applied fluence was calculated from the relation (ref. 1)

$$G = \frac{2E_0}{\pi w_0^2}$$
 (2)

where

G fluence,  $J/cm^2$ 

 $w_0$  Gaussian beam radius at optic being tested, cm

 $E_0$  average energy, J

## **Experiment Design**

To determine the influence of different procedural approaches to damage threshold testing, a ruggedness evaluation was performed to determine the significance of three procedural variables. The high and low experimental levels of the three variables evaluated in this study were

- a number of sites tested at a given fluence (10, 1)
- b number of laser pulses each spot is exposed to (200, 1)
- c diameter of incident beam (0.70 mm, 0.35 mm)

The values shown in the parentheses following each test parameter are the high and low experimental levels evaluated in this work.

Uncoated, optical grade fused silica specimens with dimensions of 5 cm  $\times$  5 cm  $\times$  0.16 cm (Esco Products, Oakridge, NJ) were employed as the test specimens in the ruggedness evaluation. Prior to testing, the optics were drag-wiped using acetone and methanol with a lint-free cloth. Additionally, the surfaces were cleaned with a static-neutralizing dry nitrogen gun before and after cleaning with solvents.

After each of the damage threshold tests was performed, optical  $(7\times-35\times)$  and Nomarski (up to  $100\times$ ) microscopy were employed to assess the presence of laser-induced damage in the test specimen. Graphs showing the probability of inducing damage as a function of the applied fluence were then prepared from the data obtained in each trial. For the trials in which 10 sites were tested at each applied fluence level, the damage threshold was determined by estimating the x-intercept through the use of regression analysis as described by Foltyn (ref. 15). In the trials using one test site for each applied fluence level, the probability of inducing damage was either 0 or 100 percent. As such, linear regression analyses were not applicable. Therefore, in these experiments the damage threshold was defined as the highest nondamaging fluence observed. Once the damage threshold was determined for all the experimental trials, the significance of each parameter under investigation was calculated over a 99-percent confidence interval as described in reference 11.

# **Experimental Results**

The damage threshold data obtained by performing tests according to the experimental design ranged from 21.5 J/cm<sup>2</sup> to 131.8 J/cm<sup>2</sup>, depending upon the experimental conditions employed in the performance of the test. A Nomarski image of a laser-induced damage site is shown in figure 4. Additionally, plots of the probability of inducing damage versus applied fluence for all trials are shown in figures 5 to 8. The damage threshold data obtained from these plots is summarized in table II.

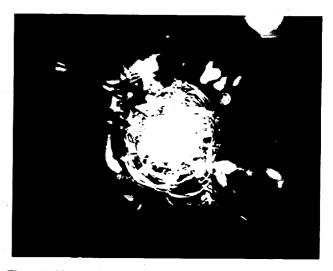


Figure 4. Nomarski microscope image of laser-induced damage in fused silica test specimen (magnification 75×).

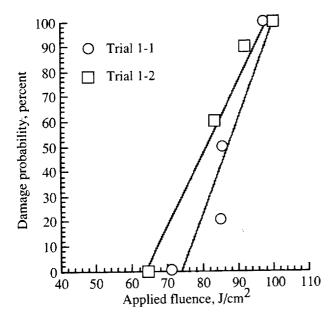


Figure 5. Damage threshold data for trials 1-1 and 1-2.

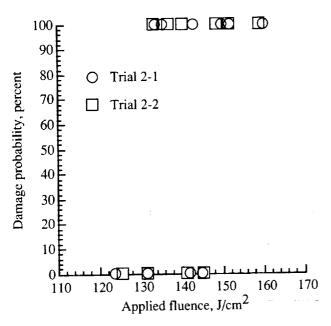


Figure 6. Damage threshold data for trials 2-1 and 2-2.

The calculated values of  $t_{N-1}$  indicate that the differences between the high and low experimental levels for the number of sites tested at each applied fluence, the number of shots to which each site was exposed, and the diameter of the incident beam were significant over a 99-percent confidence interval. The results of the ruggedness calculations are shown in table III.

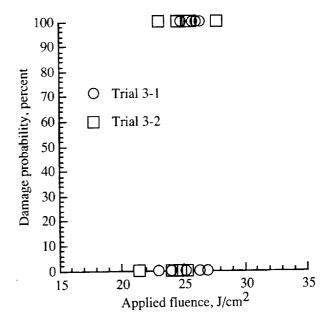


Figure 7. Damage threshold data for trials 3-1 and 3-2.

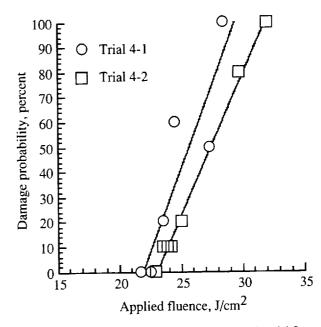


Figure 8. Damage threshold data for trials 4-1 and 4-2.

# Discussion

The first procedural variable found to influence the damage threshold was the number of sites evaluated at each applied fluence. In this study, the damage threshold data obtained by using 1 test site per fluence was found to be significantly different from the damage threshold

Table II. Damage Threshold Data Obtained by Performing Test According to Experimental Matrix

				Trial 1 threshold,	Trial 2 threshold,
Experiment	а	b	С	J/cm <sup>2</sup>	J/cm <sup>2</sup>
1	+	+	-	74.0	64.6
2	_	_	_	131.8	131.8
3	_	+	+	24.1	21.5
4	+	_	+	22.6	22.9

data obtained by using 10 test sites for each applied fluence. This finding indicates that a representative portion of an optical surface should be tested to obtain an accurate measure of the damage threshold.

In addition to the number of sites tested at each applied fluence, the damage threshold test procedure was also found to be dependent upon the number of laser pulses to which each test site is exposed. In this work, the difference between single-pulse and 200-pulse exposures was found to yield significantly different damage threshold values over a 99-percent confidence interval. This finding is in agreement with other published results, where the use of multipulse testing yielded lower damage threshold values than single-pulse tests (ref. 1).

In practice, the exposure of single test sites to single laser pulses is referred to as 1-on-1 testing, while the exposure of a single test site to multiple laser pulses is referred to as *n*-on-1 testing. One-on-1 damage threshold tests are designed to provide optics users with a measure of the initial quality of an optical component, whereas *n*-on-1 testing is more applicable to the evaluation of the lifetime performance of an optic when exposed to a continuously pulsed laser operating at a fixed repetition rate (ref. 3). Although these two procedures have different objectives, the measure of the initial quality of an optic is sometimes characterized by using multipulse exposures as described in references 6 through 9. These results

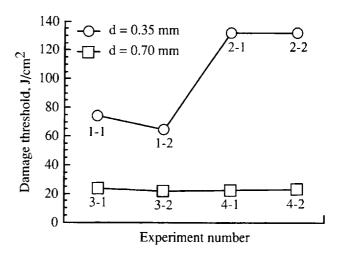


Figure 9. Difference in damage threshold obtained with beam diameters of 0.35 and 0.70 mm.

show that a significant difference in the damage threshold data is attributable to the difference between single-and 200-pulse exposures. As limited data are available on the variability of damage threshold due to the number of pulses *n* used in the procedure, initial quality determinations should be performed by using a single-pulse test protocol.

The third variable evaluated in this study was the beam diameter. In this work, a 0.35-mm beam diameter was found to yield damage threshold data significantly different from those obtained with a 0.70-mm beam diameter over a 99-percent confidence interval. At the smaller beam diameter, the average damage threshold obtained was 100.6 J/cm<sup>2</sup> with a standard deviation of 36.3 J/cm<sup>2</sup> (or 36 percent). However, when the larger beam diameter was employed, the average damage threshold obtained was 22.8 J/cm<sup>2</sup> with a standard deviation of 1.1 J/cm<sup>2</sup> (or 4.8 percent), as shown in figure 9.

The trend of increased accuracy with increasing spot size is in agreement with the previous findings of Foltyn

Table III. Results of Ruggedness Calculations on Damage Threshold Data

		Trial 1 threshold, J/cm <sup>2</sup>		Trial 2 threshold, J/cm <sup>2</sup>			
Factor	Level	Average	Effect	Average	Effect	Difference, J/cm <sup>2</sup>	t statistic (calculated)
а	10 sites/fluence	48.3		43.8			
а	1 site/fluence	78.0	-29.7	76.7	-32.9	3.2	*-14.93
ь	1 shot/site	77.2		<b>7</b> 7.4			
b	200 shot/site	49.0	28.2	43.1	34.3	-6.1	*15.39
с	0.35 mm	102.9		98.2			
c	0.70 mm	23.4	79.5	22.2	76.0	3.5	*76.95

<sup>\*</sup>Significant at 99-percent confidence interval (three degrees of freedom).

(ref. 15). In that work, the increased accuracy of the damage threshold data obtained with larger spot sizes is attributable to the increased area of the optical surface sampled by the incident beam. By employing larger beam diameters, the probability of sampling a representative portion of the optical surface is increased, thereby increasing the accuracy of the data. Conversely, the use of smaller diameter beams does not test a representative portion of the optical surface, thus inducing variability into the damage threshold data.

The identification of sources of variability in a test protocol is a critical step in ensuring the accuracy of that procedure. Otherwise, the test may yield data that falsely exaggerate differences among similar articles or artificially inflate a reported damage threshold value. Because damage threshold data are often employed as a quality control indicator by optics manufacturers, and as a means of assessing the initial quality of procured optics by laser engineers, it is necessary to identify and avoid sources of variability in the damage threshold test procedure.

### **Conclusions**

In this work, a ruggedness evaluation was performed to evaluate the influence of three procedural variables on the laser-induced damage threshold test procedure. The results of this study indicate that the damage threshold testing procedure is dependent upon the number of sites tested at each applied fluence, the number of laser pulses to which each test site was exposed, and the diameter of the incident laser beam. The combined results of this work indicate that by sampling larger portions of the optic through the use of both multiple test sites and large spot sizes, an accurate determination of the damage threshold of an optic can be made with a 1-on-1 damage threshold test protocol.

NASA Langley Research Center Hampton, VA 23681-0001 January 5, 1995

### References

- Wood, Roger M.: Laser Damage in Optical Materials. Adam Hilger, 1986.
- Thomas, Michael D.: Laser Damage Testing Boosts Quality at All Levels. *Photonics Spectra*, vol. 25, issue 11, Nov. 1991, pp. 109–112.

- 3. Seitel, Steven C.: Planning a Laser Damage Test. *Photonics Spectra*, vol. 19, Aug. 1985, pp. 84–92.
- Morgan, A. J.; Rainer, F.; De Marco, F. P.; Gonzales, R. P.; Kozlowski, M. R.; and Staggs, M. C.: Expanded Damage Text Facilities at LLNL. Laser Induced Damage in Optical Materials: 1989, NIST Special Publication 801, Oct. 1990, pp. 47-57.
- Smith, W. Lee: Laser-Induced Breakdown in Optical Materials. Opt. Eng., vol. 17, no. 5, Sept.—Oct. 1978, pp. 489–503.
- 6. Lowdermilk, W. Howard: Effects of Laser Radiation on Surfaces and Coatings. *Radiation Effects in Optical Materials*, Paul W. Levy, ed., Proc. SPIE, vol. 541, 1985, pp. 124–137.
- Guenther, K. H.; Humpherys, T. W.; Balmer, J.; Bettis, J. R.; Casparis, E.; Ebert, J.; Eichner, M.; Guenther, A. H.; Kiesel, E.; Kuehnel, R.; Milam, D.; Ryseck, W.; Seitel, S. C.; Stewart, A. F.; Weber, H.; Weber, H. P.; Wirtenson, G. R.; and Wood, R. M.: 1.06-μm Laser Damage of Thin Film Optical Coatings: A Round-Robin Experiment Involving Various Pulse Lengths and Beam Diameters. *Appl. Opt.*, vol. 23, no. 21, Nov. 1, 1984, pp. 3743–3752.
- Guenther, Karl H.; and Menningen, Roland G.: 106-µm Laser Damage of Optical Coatings: Regression Analyses of Round-Robin Test Results. *Appl. Opt.*, vol. 23, no. 21, Nov. 1, 1984, pp. 3754–3758.
- Seitel, Steven C.; and Guenther, Karl H.: Toward Standardization in Laser-Induced Damage Testing. High Power Lasers and Their Industrial Applications, Schuocker, Dieter, ed., Proc. SPIE, vol. 650, 1986, pp. 147-153.
- Arenberg, Jonathan W.; Frink, M. E.; Mordaunt, D. W.; Lee, G.; Seitel, S. C.; and Teppo, Edward A.: Correlating Laser Damage Tests. *Appl. Opt.*, vol. 28, no. 1, Jan. 1, 1989, pp. 123–126.
- 11. Standard Guide for Conducting Ruggedness Tests. ASTM Designation: E 1169-89. Volume 14.02 of 1994 Annual Book of ASTM Standards, 1994. pp. 692–697.
- 12. Natrella, Mary Gibbons: Experimental Statistics. NBS Handbook 91, U.S. Dept. Commerce, Aug. 1, 1963.
- 13. Wernimont, Grant: Ruggedness Evaluation of Test Procedures. ASTM Stand. News, Mar. 1977, pp. 13-16.
- 14. Paule, Robert C.; Marinenko, George; Knoerdel, Melissa; and Koch, William F.: Ruggedness Testing—Part I: Ignoring Interactions. *J. Res. Natl. Bur. Stand.*, vol. 91, no. 1, Jan.-Feb. 1986, pp. 3–8.
- 15. Foltyn, S. R.: Spotsize Effects in Laser Damage Testing. NBS Special Publ. 669, U.S. Dept. Commerce, 1982, pp. 368–379.

_		
_		
-		
ā		
ā		
=		
-		
- E		
-		
199		
<u>-</u>		

X.

•

REPORT	DOCUMENTATION PAGE	OMB No. 0704-0188		
gathering and maintaining the data needed, all collection of information, including suggestions	nformation is estimated to average 1 hour per r and completing and reviewing the collection of it is for reducing this burden, to Washington Hear 02-4302, and to the Office of Management and	information. Send comments reduced the design of the desig	egarding this be for Informatio	ourden estimate or any other aspect of this in Operations and Reports, 1215 Jefferson
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE February 1995	3. REPORT TYPE AND Technical Memo		VERED
4. TITLE AND SUBTITLE			5. FUNDING	G NUMBERS
A Ruggedness Evaluation o Optical Materials	of Procedures for Damage Three	eshold Testing	WU 32	23-17-04-01
6. AUTHOR(S) Matthew W. Hooker, Milfre Nina D. Tappan	d E. Thomas, Stephanie A. Wi	ise, and		
7. PERFORMING ORGANIZATION NA	ME(S) AND ADDRESS(ES)			RMING ORGANIZATION
NASA Langley Research Co Hampton, VA 23681-0001	enter		L-1743	39
9. SPONSORIING/MONITORING AGE	ENCY NAME(S) AND ADDRESS(ES)		10. SPONS	ORING/MONITORING
National Aeronautics and Sp	nace Administration			Y REPORT NUMBER
Washington, DC 20546-000			NASA	ATM-4639
	ce and Technology Corporatio Research Center, Hampton, V			
12a. DISTRIBUTION/AVAILABILITY S	TATEMENT	-	12b. DISTR	IBUTION CODE
Unclassified–Unlimited Subject Category 76 Availability: NASA CASI	(301) 621-0390			
three procedural variables of evaluated at an applied fluer sus 200 pulses), and the be	f approaches to damage threshon damage threshold data. In the nee level (1 site versus 10 sites am diameter (0.35 mm versus a 99-percent confidence interv	nis work, the difference ), the number of lase (s 0.70 mm) were all	nces between pulses a	een the number of test sites it each test site (1 pulse ver-
14. SUBJECT TERMS				15. NUMBER OF PAGES 7
Optics damage; Damage thr	eshold testing		:	16. PRICE CODE A02
17. SECURITY CLASSIFICATION	18. SECURITY CLASSIFICATION	19. SECURITY CLASSIF	ICATION	20. LIMITATION
OF REPORT Unclassified	OF THIS PAGE Unclassified	OF ABSTRACT Unclassified		OF ABSTRACT

-			
-			